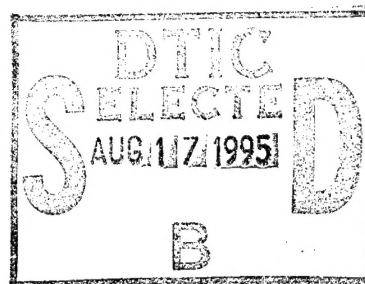# UNITED STATES ATOMIC ENERGY COMMISSION

EXPERIMENTAL WORK FOR THE DEVELOPMENT OF A PROCESS TO TREAT LUKACHUKAI ORES
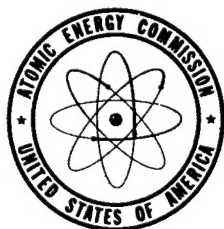
By
Galen W. Clevenger

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June 9, 1952

Atomic Energy Division
American Cyanamid Company
Watertown, Massachusetts

Technical Information Service, Oak Ridge, Tennessee

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TOPICAL REPORT ACCO-19

EXPERIMENTAL WORK FOR THE DEVELOPMENT OF A PROCESS

TO TREAT LUKACHUKAI ORES

By

Galen W. Clevenger

June 9, 1952

A B S T R A C T

Data are given for leaching ores from the Lukachukai District, Arizona with sulfuric acid or sodium carbonate. Methods are presented for a sulfuric acid extraction of both uranium and vanadium with the recovery of high grade uranium and vanadium products.

A flowsheet for recovery of uranium by sulfuric acid leaching and anion exchange of the leach solutions is recommended.

Atomic Energy Division
American Cyanamid Company
Building 421, Watertown Arsenal
Watertown 72, Massachusetts

Contract Number AT(49-1)-533

TABLE OF CONTENTS

TOPICAL REPORT ACCO-19

EXPERIMENTAL WORK FOR THE DEVELOPMENT OF A PROCESS

TO TREAT LUKACHUKAI ORES

By

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I. INTRODUCTION

Samples of ore from the Lukachukai District, Arizona were submitted to this labora-
tory by the Division of Raw Materials of the U. S. Atomic Energy Commission for the
purpose of developing a flowsheet and preparing estimates for a plant to recover
uranium and vanadium. This report summarizes the experimental work conducted in
the laboratory on which our recommendations were based.

Considerable tonnage of these ores are of relatively high lime content and for this
reason are not amenable to the conventional salt roast process. The vanadium con-
tent of much of the ore is sufficient to make consideration of its recovery
attractive.

However, while the uranium is easily leached from the ore with sulfuric acid or
soda ash the vanadium is not. Good vanadium extractions were only obtained by
relatively long contact with hot concentrated acid. This approach for vanadium
recovery is included along with the data on uranium recovery in this report. Dur-
ing the course of this work favorable results using a salt roast on a sulfated
high lime ore were reported* and it was decided to investigate salt roasting of
the residue following uranium recovery with sulfuric acid. This procedure has
given very promising results and is expected to be more economical than treating
the residue with strong acid. The salt roasting of these residues for vanadium
recovery will be the subject of a future report.

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*K.B. Brown, F.J. Hurst, J.M. Schmitt, Y-826 Recovery of Uranium and Vanadium
From High Lime Ores - 11/9/51
Carbide & Carbon Chemicals Co. - Y-12

II. SUMMARY & CONCLUSIONS

The uranium content of the ore may be readily leached with either sulfuric acid or sodium carbonate. Some vanadium (10-20 per cent), presumably that associated with the uranium as carnotite, is also easily leached. The remainder of the vanadium is more difficultly soluble and requires prolonged contact with hot sulfuric acid. Best results were obtained when the ore was mixed with concentrated acid, baked at low temperature (100-200°C) and leached with hot water.

For uranium extraction a sodium carbonate requirement of 100 to 150 pounds per ton was indicated. For sulfuric acid leaching reagent consumption depended on the lime content of the ore and for the samples tested was between 200 and 250 pounds of H_2SO_4 per ton of ore. For vanadium extraction from this residue about 400 pounds of additional acid were required.

The uranium from the acid leach liquor was recovered in a precipitate assaying about 90% U_3O_8 using anion exchange resin. Vanadium has been recovered from acid leach liquors as red cake by oxidizing and adjusting the pH of hot solutions.

The flowsheet recommended for recovering uranium from this ore consists of a cold sulfuric acid leach at pH 1.0 - 1.1 (200-250 pounds H_2SO_4 per ton of ore for samples tested) followed by adsorption of the uranium in solution on an anion exchange resin; elution of the uranium from the resin with hydrochloric acid, and precipitation of a high grade product with ammonia.

Vanadium may be recovered from the uranium leach residues by use of a low temperature (100-200°C) bake with additional sulfuric acid. The vanadium extracted in the uranium leach procedure (10-20 per cent of the total) would be combined with this or, in the case of ores too low in vanadium to warrant the additional leaching step, precipitated directly by heating and oxidizing.

No recommendations are made concerning the vanadium section of the plant at this time. An alternate to vanadium recovery from the residue by acid baking is to salt roast. In either case the vanadium section would be an entirely separate unit with no interchange of solutions other than a bleed of vanadium from the uranium circuit.

III. RECOMMENDED FLOWSHEET

The ore was found to be amenable to leaching with both soda ash and sulfuric acid. A flowsheet using sulfuric acid with uranium recovery by anion exchange has been chosen for the following reasons:

1. Uranium minerals are more readily soluble in sulfuric acid than in sodium carbonate.
2. Leaching and precipitation can be carried out at ordinary temperatures and there is no need for process steam.
3. Filtration is more favorable. With soda ash leaching there was a classification of slimes from the pulp which would make a difficult liquid-solids separation and recovery of water soluble values. The pulp from an acid leach is homogeneous and lends itself to displacement washing.
4. Precipitate grade is considerably better than that obtained with a conventional yellow cake precipitation from carbonate solution.

In choosing the flowsheet it has been assumed that the ore available for treatment in the plant will be similar to the sample submitted to this laboratory for testing. This is particularly important with regards to U_3O_8 and lime content. Recovery of vanadium from the ore has been investigated and it has been determined that good extractions of this element can be obtained by a concentrated sulfuric acid treatment. This, however, dissolves other impurities from the ore which interfere with subsequent precipitation of uranium. Experiments have been made with various flowsheets and it has been determined that vanadium can best be recovered from the residue following uranium extraction rather than attempting to extract both metals simultaneously from the ore and effect a subsequent separation.

The plant has been designed primarily for uranium recovery with consideration given the possible future recovery of by-product vanadium. The vanadium recovery unit is considered as a separate plant which would have to stand on its own merits. The proposed leaching procedure for uranium extracts 10 - 15% of the vanadium from the ore. This amounts to 2 - 3 pounds of V_2O_5 per ton of ore. This vanadium is in solution and can probably be recovered simply by heating and oxidizing. While this has been done in a number of laboratory tests, results have been difficult to duplicate. If this vanadium cannot be precipitated as a "red cake" of satisfactory grade a sludge can be precipitated with alkali and stocked for future treatment. From a cursory investigation of various operating plants it has been observed that the physical conditions existing during the "red cake" precipitation are important. Consequently, plant conditions are probably difficult or impossible to duplicate on a small scale.

If the ore originally assayed 1% V_2O_5 about 14 pounds additional V_2O_5 could be extracted with 400 pounds of H_2SO_4. It is not possible to say at this time that this vanadium could be recovered in a satisfactory "red cake". Ore too low in vanadium for economic recovery is the logical material to treat in the plant, initially.

If a satisfactory vanadium recovery process is worked out in the laboratory, it can be added to the existing flowsheet without interruption of uranium production.

Should it become necessary to treat high vanadium ores, the residues can be stocked separately to facilitate their return to the plant for possible future recovery of vanadium.

Discussion of Flowsheet

The plant has been divided into three sections. These are drying and grinding, leaching and filtration, and ion exchange and precipitation. The various sections will be discussed separately.

A. Drying and Grinding

The plant feed is assumed to have been sampled and minus one inch. The ore will be delivered to a feed hopper and elevated to a 600 ton storage bin. From this bin it will go either through a dryer or directly to a 3/8 inch screen in closed circuit with a cone crusher. The minus 3/8 inch material will be stored in a second 600 ton bin. From here the ore goes to a rod mill in closed circuit with vibrating screens where it is reduced to minus 35 mesh. The grinding plant flow-sheet is shown in figure 1 on the following page. Three 600 ton storage bins have been located in the circuit to permit a three day shut down of any major piece of equipment without interrupting production.

Dry grinding has been selected in preference to wet so that adequate washing of the leach residues will be possible without making large solution discards necessary. A bleed from the uranium leaching circuit is contemplated but it will be regulated to keep the vanadium assay at a maximum. This solution will be treated for vanadium recovery or possibly be used as a leaching solution should a future plant be built for vanadium recovery from the uranium leach residues.

Minus 35 mesh is below the minimum size necessary for satisfactory leaching extraction, but coarser grinding would result in sand classification in leaching agitators and filters.

All grinding for a 24 hour period is to be done in one 8 hour shift. This requires a larger installation but permits the use of considerably less labor per ton of ore ground than if a 24 hour operation were used. It further allows for expansion of production simply by operating more than one shift per day. The large power demand is balanced by leaching only on the other two shifts.

B. Leaching and Filtration

This operation will be carried out on a two shift per day basis thus effecting a saving in labor. The connected horsepower here is about 75% of that in the grinding section which will help equalize the power load particularly if the leaching is done on the night shifts.

Ore, acid and returned effluent from the ion exchange section is to be fed continuously to three mechanically agitated tanks connected in series. Two of these will be for leaching and provide for a minimum of 6 hours leaching time. The third tank will serve both for leaching and as a surge tank ahead of the filters. The continuous leach will allow a maximum time for acid addition which should minimize foaming due to CO_2 evolution.
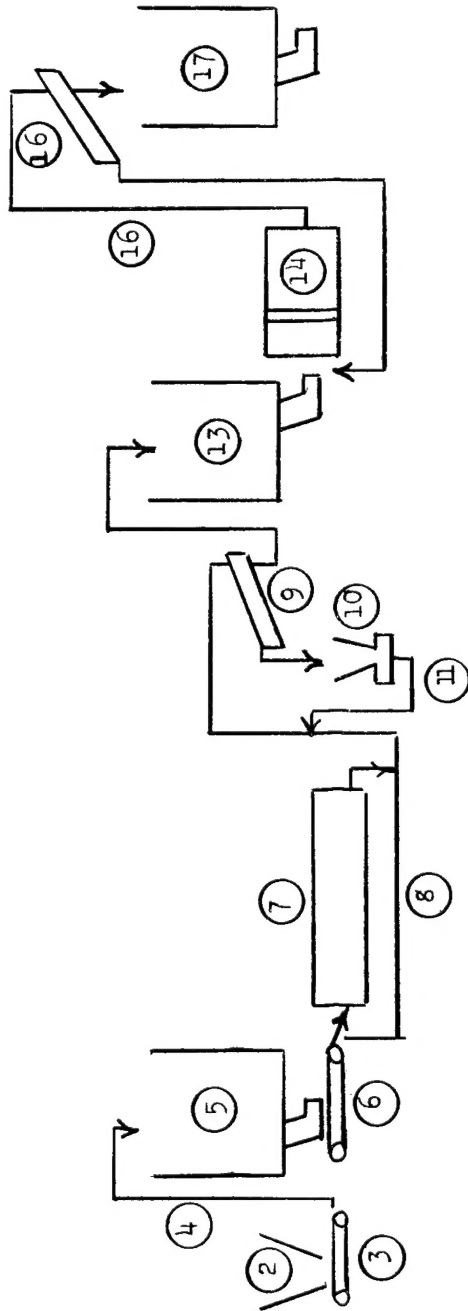
Figure 1: Grinding and Drying Flowsheet – 200 Tons per 8 Hours

B. Leaching and Filtration (Cont'd)

Filtration is to be done on Moore filters. Careful consideration has been given
to filter selection and because of the non-uniformity of the pulp and the need for
proper washing of the residues the Moore type filter was chosen. With this filter
the operator will have maximum flexibility of filtering and washing which would
not be possible with continous filters. Residue from the Moore filter is pumped
to waste or if high enough in vanadium to suitable storage ponds. All water enter-
ing the plant will be as wash water either on the ion exchange columns or at the
Moore filter to be used in the first, or strong wash tank.

Storage for one days' production of filtrate is provided. This filtrate, after
clarification in filter presses, is sent to a second storage tank of equal size
from which it is fed to the ion exchange columns. The flowsheet for this section
of the plant is shown in Figure 2 on the following page.

C. Ion Exchange and Uranium Precipitation

This section of the plant will run on a 24 hour per day basis. Clarified Moore
filtrate is fed through the column until the resin becomes loaded with uranium to
the point where less than 95 - 99% is being removed. The solution coming from
the column (effluent) will return to the leaching section except for a portion
low in uranium which will be bled for vanadium precipitation. Then the feed solu-
tion is switched to another column. While this column is being loaded with uranium
the column already loaded is washed with water and the uranium eluted with an
acidified chloride solution. This procedure will alternate from one column to
another with a total of five complete cycles per day. The chloride eluting solu-
tion is neutralized with ammonia in mechanically agitated tanks and the uranium
precipitate filtered on plate and frame filter presses.

The neutral chloride eluting solution is acidified with hydrochloric acid in a
mechanically agitated tank and is ready for re-use in the columns. The uranium
precipitate is then calcined in a gas fired hand rabbled furnace and is ready for
shipment.

The single column operation described, while probably more costly in reagent con-
sumption is suggested in preference to multiple column saturation loading because
of difficulties that have been encountered in recovering uranium from columns
containing higher concentrations of uranium. One extra column has been provided
for flexibility of the ion exchange section. The flowsheet for the above is shown
in Figure 3 on page 8.
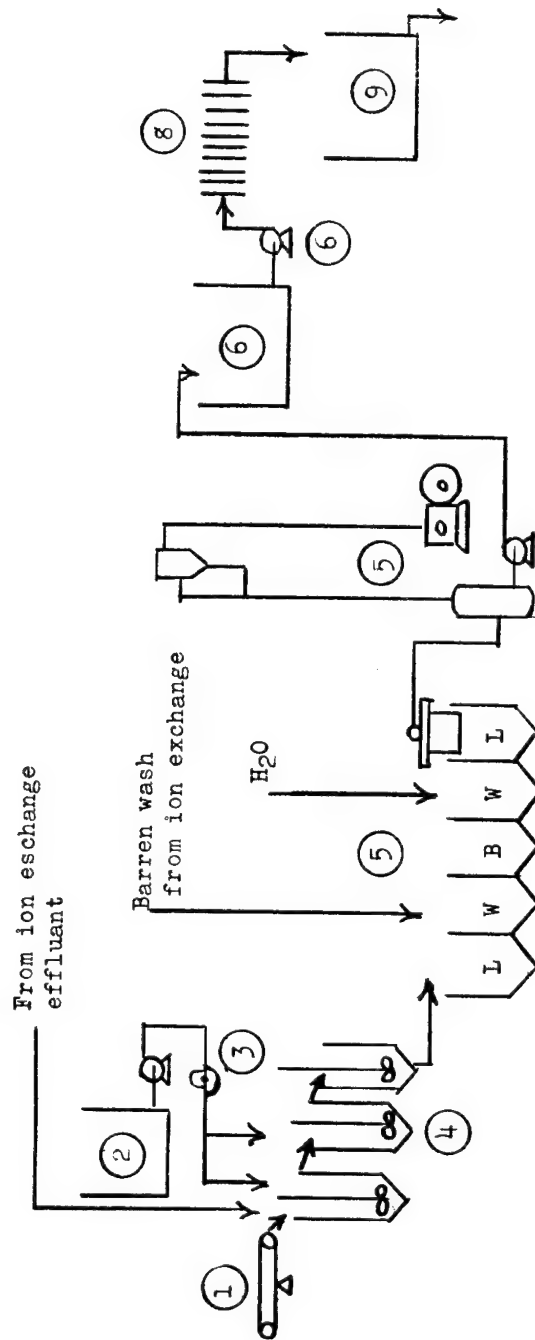
Figure 2: Leaching and Filtration Flowsheet - 200 Tons Ore Per 16 Hours

Leach Soln.

H$_2$O

To
Leach

To
Moore Blow
or V Precipitation
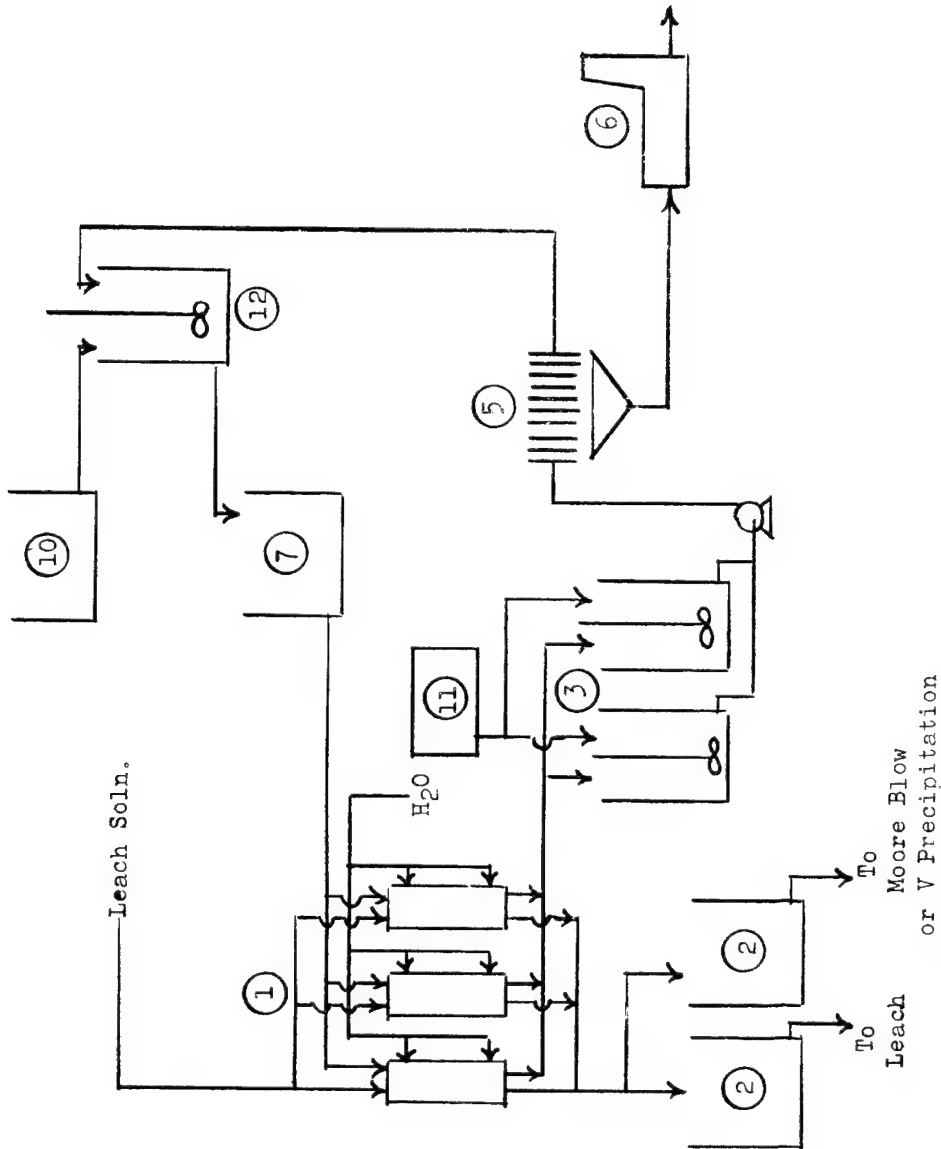
Figure 3: Ion Exchange and U Precipitation

Equipment & Labor Requirements

Tables I to VI appearing on the following pages give estimates of labor, power and equipment requirements for the 200 ton per day plant.

Table I

Operating Labor Requirements

Department	Classification	Man Shifts Per Day
Drying and Grinding		
	Trucker	1
	Operator	1
	Helper	1
Leaching and Filtration		
	Leacher	2
	Helper	2
	Filterman	2
	Helper	2
	Helper	2
Ion Exchange and Precipitation		
	Operator	3
	Helper	3
	Furnaceman	1
Maintenance		
	Mechanic	1
	Mechanic	1
	Mechanic	1
	Mech. Helper	1
	Mech. Helper	1
	Electrician	1
Common Labor		
	Misc. Including Watchmen	8
TOTAL		34

Table II

Laboratory, Office and Supervision Labor Requirements

Department	Classification	Number
Laboratory		
	Plant Metallurgist	1
	Chief Chemist	1
	Chemist	2
Office		
	Chief Clerk & Purch. Agent	1
	Clerk	3
Supervision		
	Superintendent	1
	Shift Boss	4
	Master Mechanic	1
TOTAL		14

Table III

Electric Power Requirements

Drying and Grinding	Horse Power
Rod Mill	250
Dryer	60
Cone Crusher	60
Conveyers etc.	41
Screens	9
Miscellaneous	30
Total	450

Leaching and Filtration	
Leach Agitators	45
Vacuum Pumps	120
Air Blowers	40
Pumps	35
Moore Filter Cranes	60
Miscellaneous	30
Total	330

Ion Exchange and U Precipitation	
Precipitation Agitators	15
Pumps	26
Miscellaneous	10
Total	51

Table IV
Plant Equipment

Drying and Grinding

Item

1. 1 1½ cu. yd. type H. M. gasoline driven Hough Payloader

2. 1 10' x 10' steel loading hopper - 60° sides (5500 lbs)

3. 1 36" x 6' apron feeder - 1/4" thick steel pans 6" high sides variable speed drive from 5' - 20' per min. 5 H.P. Motor (4000 lbs)

4. 1 24" x 125' belt conveyor, inclined 18° with roller bearing idlers, 5 ply 32 ozs. conveyor belt, chain and sproket drive, 15 H.P. 125 RPM class, 3 gear reducing motor (39,600 lbs) complete with magnetic head, pulley and 1½ Kw M.G. set

5. 1 Steel ore bin 24' dia. x 35' for 600 T - 1" ore (48,000 lbs)

6. 1 24" x 25' Merrick Feed-o-Weight with 2 H.P. motor (7000 lbs)

7. 1 8' x 70' Rotary dryer with 60 H.P. TEFC high torque motor and Buffalo No. 110, Type MW 50,000 CFM fan with bag type D-18-32-240 dust collector (185,000 lbs)

8. 1 Belt conveyer 24" x 180' with idlers head and tail puller and drive, last 100' inclined at 20° 5 H.P. drive (16,000 lbs)

9. 1 Single deck horizontal vibrating screen 3' x 8' with 3/8" mesh opening, 3 H.P. motor (7100 lbs)

10. 1 3' Symons standard cone crusher with 60 H.P. high torque motor (24,400 lbs)

11. 1 Belt conveyor 24" x 70' with idlers and pulleys, inclined at 12° with 2 H.P. motor (7,100 lbs)

12. 1 Belt conveyor 24 x 76 complete with 3 H.P. motor (6,600 lbs)

13. 1 Ore storage bin 24' dia. x 35' (48,000 lbs)

14. 1 6' x 14' Marcy rod mill with drive accessories except motor and controls (97,000 lbs)
   1 Rod charge (74,000 lbs)
   1 250 H.P. 3 phase 60 cycle wound rotor motor (6000 lbs)
   1 Starter and controller (1090 lbs)

15. 5 AC Utah Electromagnetic vibrating screens totally enclosed

16. 1 35 ft. chain belt elevator, 3 H.P. drive 8" x 5" buckets (6800 lbs), 3 H.P. Falk drive (6800 lbs)

17. 1 Fine ore bin 24' dia. x 35' (48,000 lbs)

18. Miscellaneous screw conveyors and drives - 10 H.P.

19. Dust Collection - 10 H.P.

Table V

Plant Equipment

Leaching and Filtration

Item

1.	1	24" x 25' Merric Feed-o-Weight (7000 lbs) - 2 H.P. motor
2.	1	10,000 gallon steel storage tank for sulfuric acid with $1\frac{1}{2}$" pump and 5 H.P. motor
3.	1	Disc and bucket sulfuric acid feeder - with 1 H.P. motor (1500 lbs)
4.	3	14' x 14' Steel tank lined with 3/16" rubber and 9" of acid brick
	3	Falk vertical motor ducers No. 15562R - 37 RPM - 15 H.P. each
	3	Supports and agitators
5.	1	Moore filter unit Tanks (10) 12' x 20' x 6' deep rubber covered steel
	4	Filter baskets total area 8000 sq. ft. with saran cover
	2	20 Ton electric cranes 30 H.P.
		Piping valves and traps
	4	18" x 7" - 720 CFM vacuum pumps 30 H.P. each
	2	American blowers - 6-28-F - 1000 CFM - 2 PSI - 20 H.P. each
	3	$1\frac{1}{2}$" Filtrate pumps 3 H.P. each
6.	1	30' dia. x 13' wood stave tank
7.	2	100 G.P.M.-105 ft. head olivite pumps $2\frac{1}{2}$" inlet - 2" outlet 10 H.P. each with TEC motor
8.	2	Filter presses 36" - 27 frames yellow pine - hydroclosing
9.		Miscellaneous piping valves launders etc.
10.	1	30' dia. x 13' wood stave tank

Table VI

Plant Equipment

__Ion Exchange and U Precipitation__

__Item__

1. 3 Rubber lined steel columns 5'6" x 14' complete with distributors, collectors, pipe fittings and controls

2. 2 30' Dia x 13' wood stave tank

3. 2 12' Dia x 15 wood stave tank

    2 Falk Vertical Motorducers 5 H.P.

    2 Supports and agitators

4. 2 100 G.P.M. - 105 ft. head Olivite pumps $2\frac{1}{2}$" inlet 2" outlet, 10 H.P. each with motor

5. 2 36" 27 Plate Filter presses, yellow pine - Hydroclosing

6. 1 Calcining furnace - Hand rabbled gas fired 6' x 12'

7. 1 25' Dia x 12' wood stave tank

8. 2 Olivite Pumps 50 G.P.M. - 50 ft. head $1\frac{1}{2}$" outlet 2" inlet with 3 H.P. motor

9. 1 Gasoline lift truck

10. 1 HCl Storage Tank Rubber Line 10' x 10'

11. 1 NH_3 Storage Tank 10' x 10'

12. 1 12 Dia x 15 wood stave tank

    Falk Motorducer 5 H.P.

    Supports and Agitator

13.   Piping and Fittings

14.   266 cu. ft. of Resin at $60 per cu. ft.

IV. PRELIMINARY INVESTIGATIONS

Six 83 pound samples of Lukachukai ore were shipped to us from Grand Junction, Colorado on March 29, 1951, and were identified in letters from Mr. J.L. Chapman in the following manner.

Table VII

Lot No.	% U_3O_8	% V_2O_5	% $CaCO_3$
6144	0.32	0.96	9.9
6184	0.48	0.83	9.1
6127	0.30	0.95	8.9
6128	0.25	0.96	8.2
6158	0.27	0.85	9.4
6166	0.25	0.73	10.5

It was stated that each sample represents 60 to 70 tons of ore shipped by F. A. Sitton, Inc. from the Lukachukai Mountain mining district, Apache County, Arizona; Lots 6127 and 6158 were from Cove Mesa No. 4. The others were from Cove Mesa No. 1.

The ores are of a sandstone type, composed of quartz cemented with calcite, carnotite, clay and vanadium bearing micaceous clay minerals. Accessory minerals observed were principally hematite, magnetite, zircon and tourmaline; organic matter was also present. The major uranium mineral observed was carnotite, with tyuyamunite also being present. Although part of the vanadium was present in association with the carnotite, the major part of it was in the fine hydrous mica clay fraction. Screen analyses of the minus 20 mesh ore charges show approximately 85 per cent by weight minus 65 mesh and about 15 per cent minus 270 mesh. Assays on various size fractions show that 50 to 60 per cent of the uranium and vanadium content are concentrated in the minus 270 mesh fraction. Table VIII gives the results of partial chemical analyses on the six samples.

Table VIII

Lot No.	Cyanamid Sample No.	% U_3O_8	% V_2O_5	% SiO_2	% CaO	% Al_2O_3	% Fe
6127	17-1	0.328	1.09	78.9	4.70	3.2	0.76
6128	17-2	0.270	1.13	78.2	4.48	3.9	0.78
6144	17-3	0.335	1.09	76.1	5.53	2.9	0.74
6158	17-4	0.300	1.13	77.2	5.08	2.0	0.84
6166	17-5	0.261	0.91	78.1	5.47	2.9	0.69
6184	17-6	0.330	1.05	78.2	5.08	2.5	0.79

The second shipment of Lukachukai ore (one ton) to this laboratory was received January 7, 1952. It appears similar to the original shipment except for a somewhat higher lime content. This material has been designated by the laboratory as Sample 17-7 and assays as shown in Table IX which follows on the next page.

Table IX

Assay of Sample 17-7 Ore

U_3O_8	0.290%
V_2O_5	1.03 %
CaO	6.4 %
Fe	1.1 %
SiO_2	74.4 %
Al_2O_3	2.6 %
P_2O_5	0.1 %

All samples were received in good condition and were reduced to minus 20 mesh by crushing and screening. This minus 20 mesh product was used in the test work without further reduction.

Screen analysis of the minus 20 mesh ores follow:

Table X

Screen Analysis of Minus 20 Mesh Ore

Tyler Mesh	17-1 Ore	17-2 Ore	17-3 Ore	17-4 Ore	17-5 Ore	17-6 Ore	17-7 Ore
	%	%	%	%	%	%	%
+35	6.62	6.96	5.86	6.21	6.47	5.24	5.19
-35+65	8.70	8.11	8.13	8.52	8.96	7.83	10.44
-65+100	9.09	8.86	10.50	8.50	9.10	10.23	20.46
-100+150	38.28	42.69	39.51	38.55	39.81	42.96	(
-150+200	14.60	13.95	12.48	15.08	13.77	12.93	(38.91
-200+270	5.59	4.68	4.76	6.41	5.83	4.82	5.17
-270	17.12	14.75	18.76	16.73	16.06	15.99	19.83
	100.00	100.00	100.00	100.00	100.00	100.00	100.00

V. TEST WORK

Leaching Tests

Both sulfuric acid leaching and sodium carbonate leaching tests were made on the six samples. Sulfuric acid leaching was at room temperature, 50 per cent solids, and 3-hour agitation period. Sodium carbonate leaching was at 70 to 80°C., 50 per cent solids, and 5-hour agitation period. All leaching tests were conducted on ore crushed to minus 20 mesh. The results of these tests are summarized in Table XI.

Table XI

Sample	Lbs./Ton Added		% U₃O₈		% Extraction	
	H_2SO_4	Na_2CO_3	Feed	Residue	U_3O_8	V_2O_5
17-1	200	--	0.263	.020	92.1	13.9
17-1	300	--	0.298	.005	98.7	16.6
17-1	150	--	0.277	.021	92.2	11.2
17-1	--	200	0.277	.007	97.4	13.1
17-1	--	100	0.330	.045	86.4	11.5
17-2	200	--	0.272	.012	95.4	18.1
17-2	300	--	0.253	.004	98.3	18.7
17-2	150	--	0.297	.012	95.8	16.8
17-2	--	200	0.251	.009	96.3	18.3
17-2	--	100	0.258	.034	86.8	14.9
17-3	224	--	0.344	.014	96.9	12.0
17-3	300	--	0.371	.007	98.1	15.1
17-3	150	--	0.315	.258	15.4	3.3
17-3	--	200	0.331	.016	95.2	11.7
17-3	--	100	0.327	.041	87.4	11.0
17-4	218	--	0.251	.011	95.5	9.4
17-4	300	--	0.262	.005	98.2	15.8
17-4	150	--	0.253	.033	86.6	8.5
17-4	--	200	0.268	.006	95.9	10.6
17-4	--	100	0.266	.018	93.2	8.5
17-5	216	--	0.262	.019	92.3	10.9
17-5	300	--	0.263	.006	98.3	18.7
17-5	150	--	0.248	.236	1.6	0.0
17-5	--	200	0.243	.010	95.9	13.1
17-5	--	100	0.245	.021	91.4	10.1
17-6	200	--	0.302	.020	93.1	13.2
17-6	300	--	0.357	.006	98.3	15.1
17-6	150	--	0.346	.020	94.1	10.0
17-6	--	200	0.316	.010	96.8	11.1
17-6	--	100	0.315	.027	91.4	10.1
17-Comp.	300	--	0.255	.008	96.8	11.2
17-Comp.	300*	--	0.275	.004	98.7	25.9

* Hot leach, 80°C.

Leaching tests on the 17-7 ore run for four hours at room temperature and 50 per cent solids indicated that the acid requirements would be about 250 pounds of H_2SO_4 per ton of ore. Figure 4 summarizes the results of these tests.
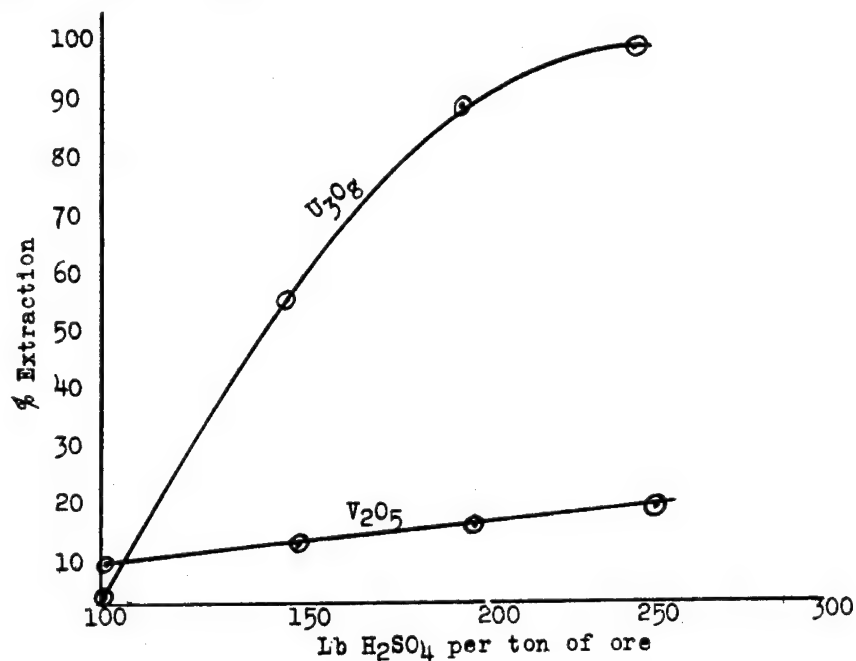
Figure 4. Leaching Tests of Sample 17-7 Ore

Precipitation of Leach Solutions

Precipitation of the carbonate leach liquor by acidification to pH 5.0 produced a precipitate assaying 65 per cent U_3O_8 and containing 95 per cent of the uranium in the liquor.

Stepwise neutralization of the acid leach liquor with NH_3 produced the following results.

Table XII

Product	Pptn. pH	% Assays		% Dist.	
		U_3O_8	V_2O_5	U_3O_8	V_2O_5
1st Ppt.	4.0	41.3	13.5	33.5	22.2
2nd Ppt.	5.1	44.2	26.8	50.4	62.6
3rd Ppt.	8.0	26.2	13.2	14.6	15.1
Comb. Ppt.	-	38.2	19.5	98.5	0.1
Barren Soln.	-	.0016	nil		

Ion Exchange on Acid Leach Liquors

A leach liquor derived from a leach of the composite sample with 300 pounds H_2SO_4 per ton was put through a column of IRA-400 resin at a retention time of 10 minutes. The feed solution assays were 1.48 grams U_3O_8 and 0.79 gram V_2O_5 per liter; pH was 0.8. Breakthrough occurred after 16.4 column volumes were passed through the column; loading at that point was 24 mgs. U_3O_8 per ml wet settled resin.

The resin column was then washed and eluted with a $0.9N$ $NH_4Cl:0.1N$ HCl solution using a 10 minute retention time. Ignition of a precipitate, produced by neutralizing the eluate with NH_3 to pH 7.0, gave a product that assayed 87.3 per cent U_3O_8.

In another test using a leach liquor at pH 1.1 the resin loading increased to over 50 mgs. U_3O_8 per ml wet settled resin. Figures 5 to 7 on the following pages show the effect of various flow rates on the performance of resin columns with leach liquor at pH 1.1. The loading of these columns proceeded past the point where uranium in the effluent started to increase from the minimum value (breakthrough) to the point where the assay of the solution entering the column was the same as that leaving (saturation). Elution of these saturated columns with $1N$ HCl: $0.9N$ NH_4Cl was incomplete. Continuation of the elution with $1.0N$ HCl resulted in the removal of the remaining uranium. Figure 8 on page 22 shows the elution of one of these saturated columns. Elution of columns loaded to breakthrough was complete with $0.1N$ acid. The cause of the difficulty in eluting the saturated columns is not certain, but it is thought that the solution is so highly concentrated that uranium precipitates (possibly as a phosphate) in the resin bed and is only soluble in relatively strong acid.

In Figures 5 to 7 the uranium adsorbed on the column is the area between the dotted lines and the curves. Increasing the retention time from 2 minutes (Fig. 5) to ten minutes (Fig. 7) more than doubled the quantity of uranium adsorbed by the resin at breakthrough. The additional capacity gained by continuing to saturation (at ten minute retention time) was only about 20% of the total. This added 20% capacity is probably not economic to go after if stronger acid is necessary to elute it.
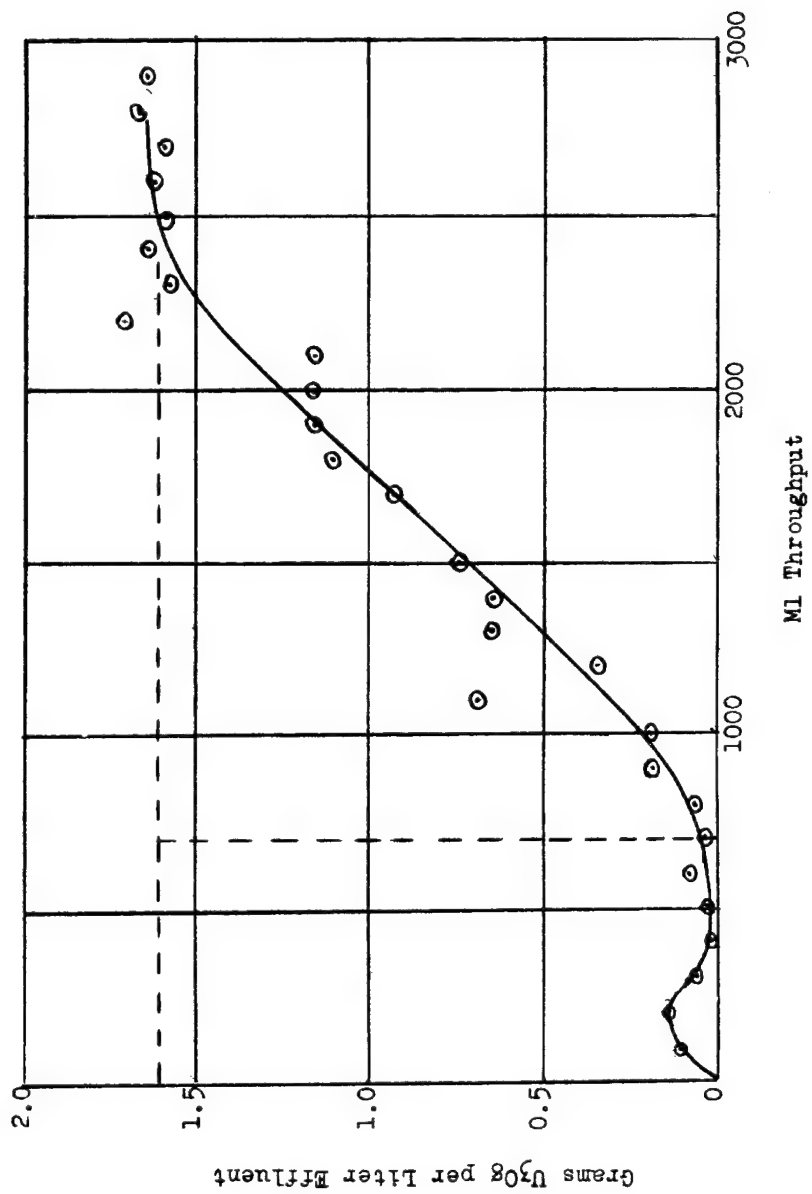
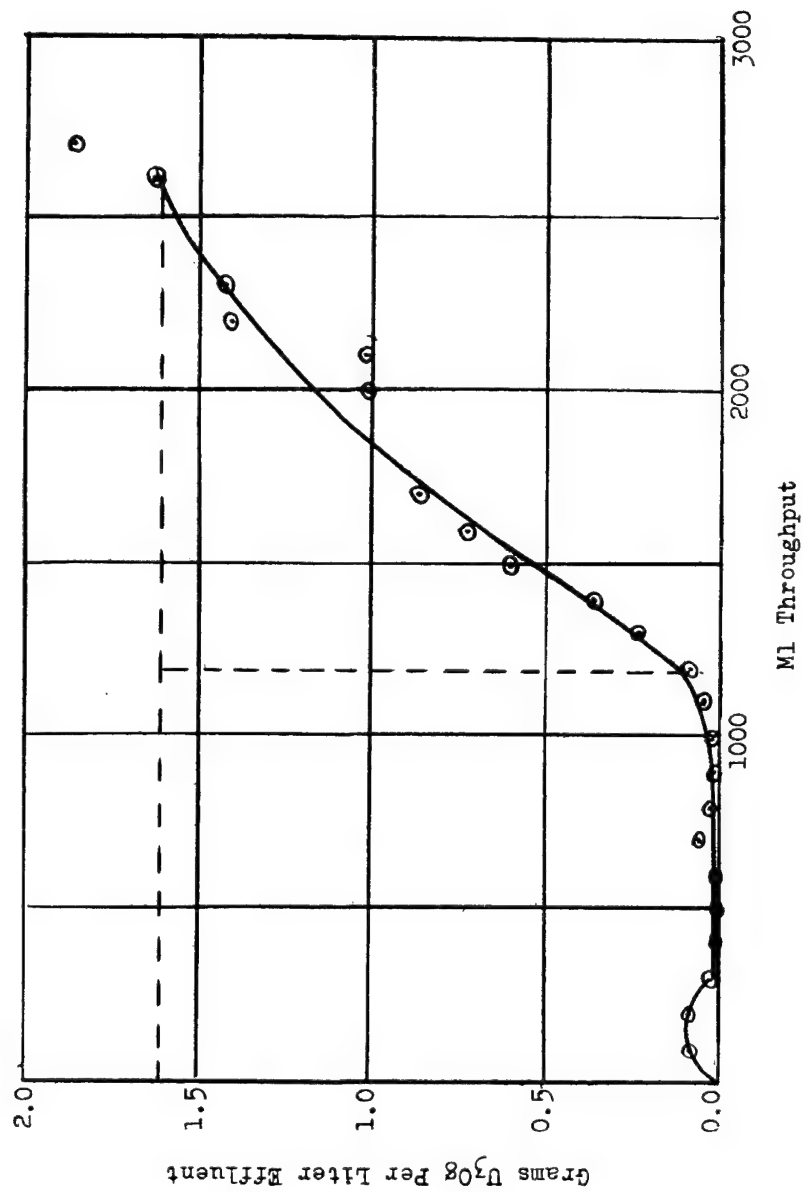Figure 5: Saturation Curve Two Minute Retention Time pH 1.1

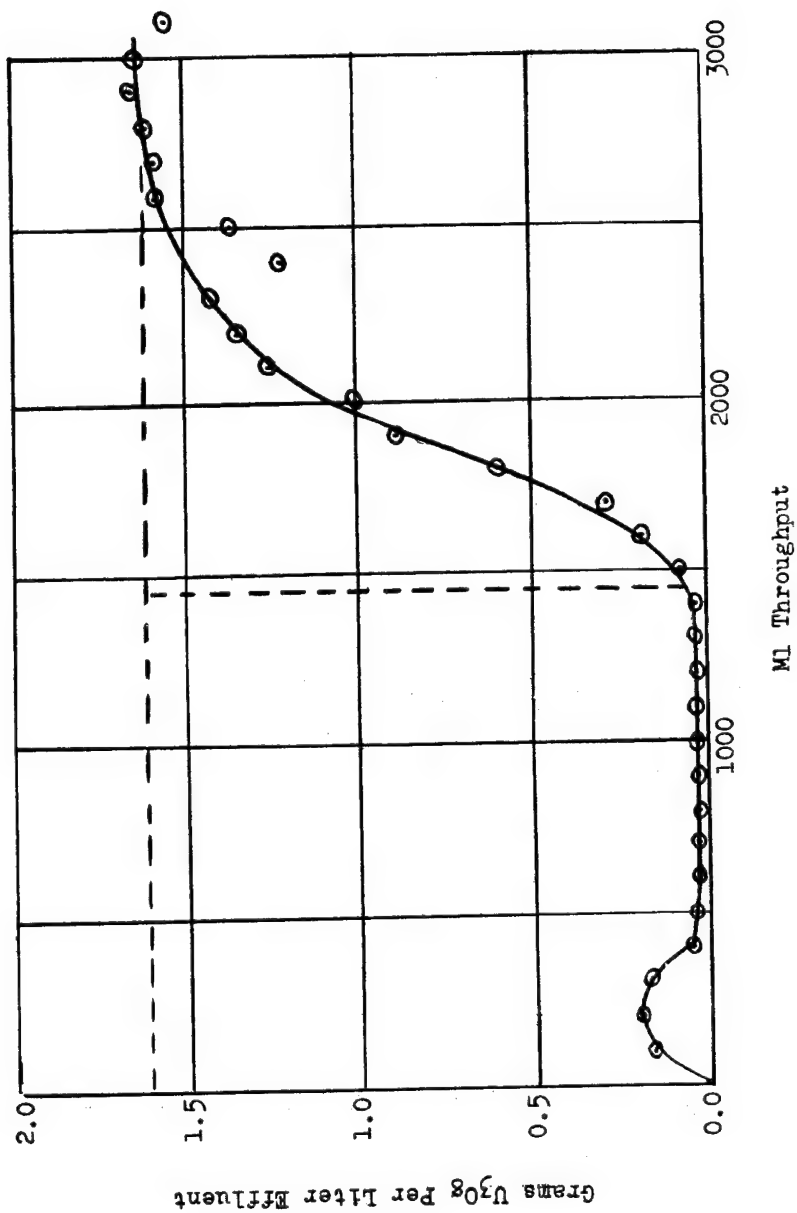Figure 6: Saturation Curve Four Minute Retention Time pH 1.1

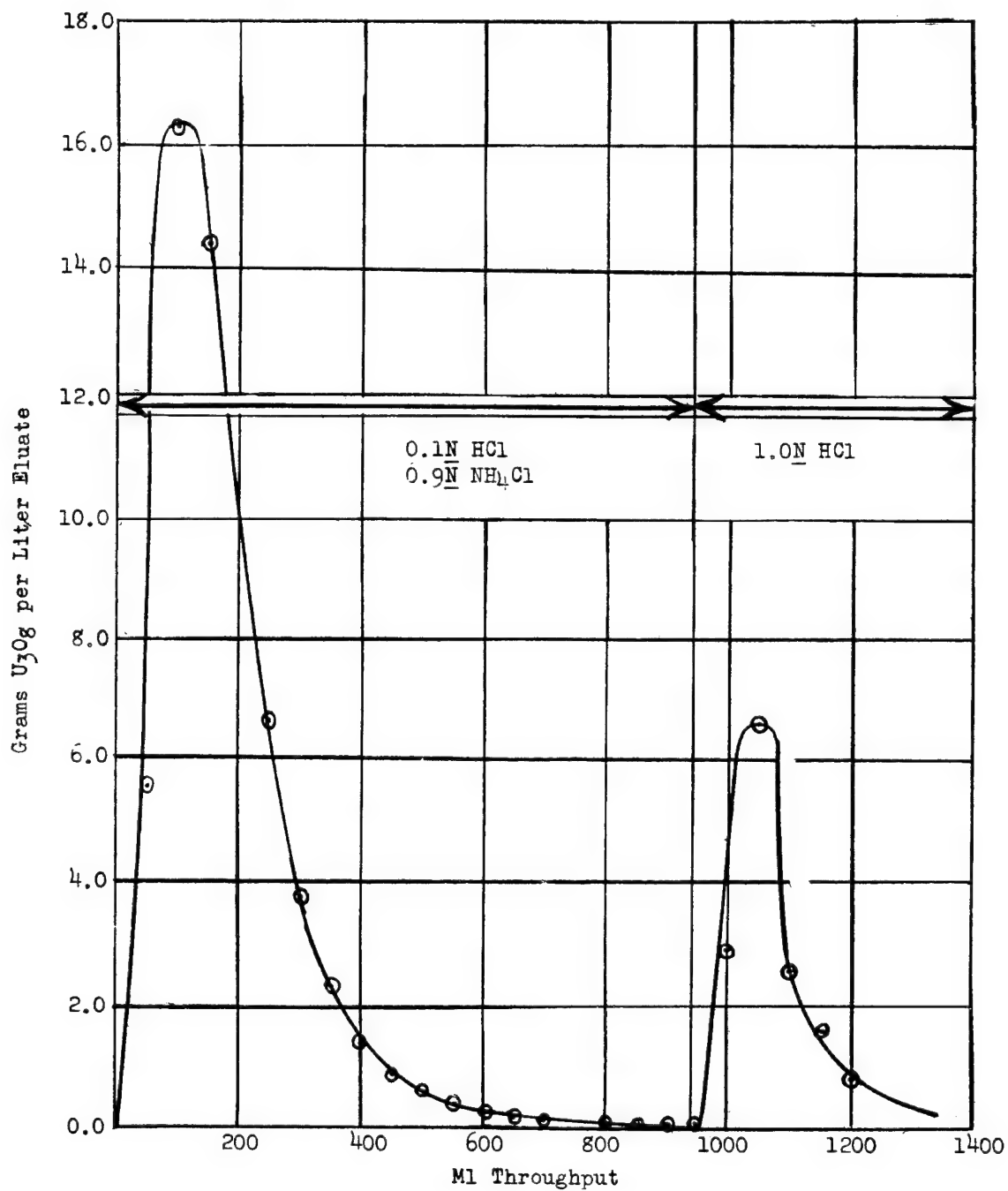Figure 7: Saturation Curve Ten Minute Retention Time pH 1.1

Figure 8: Elution Curve Eight Minute Retention Time

It is interesting to note in Figure 8 which is the elution following the ten minute
retention time loading that the additional uranium eluted with strong acid is also
about 20% of the total. This suggests that making the cutoff loading more nearly
approach the saturation loading by increasing the retention time beyond ten minutes
might result in elution difficulties with 1.0N HCl. This was not investigated as
loading times longer than ten minutes were not being considered in order to keep the
resin inventory at a minimum. These preliminary tests were used as a basis for es
tablishing conditions for ion exchange of solutions made in tests of various flow-
sheets described later in this report. Since the resin capacity increases with pH
(more than twice as great at pH 1.1 than at pH 0.8) control of this factor is impor-
tant. Maximum resin loading is desirable because the more uranium that can be put
on the resin, the smaller is the unit cost of reagents (HCl and NH$_3$) to remove and
precipitate it. In a plant operation on this ore there would undoubtedly be day to
day, if not shift to shift, variations in lime content of the feed. This would make
close pH control of the leach pulp difficult. For optimum extraction and resin load-
ing the pH should be between pH 1.0 and 1.2. Higher pH will result in a falling off
of leaching extraction which would be serious. Lower pH would cause the uranium ca-
pacity of the resin to decrease. In addition to the reason already mentioned this
decrease in capacity would be undesirable because of the possibility of the capacity
of the resin falling below that required for complete removal of the uranium from solu-
tion. This condition might also come about due to fluctuations in the uranium assay
of the feed to the ion exchange column which in this type of operation could conceivably
be plus or minus 50%. So that the operation can be flexible the column effluent should
be recirculated to the leaching section in order that the column can be operated at
maximum capacity without danger of losing uranium. This recirculation of solution
causes the vanadium in solution to build up as it is apparently in the plus four valence
state and not adsorbed on the resin. This vanadium can be bled from the system at a
relatively high concentration and precipitated as "red cake" or sent to a vanadium leach-
ing section to be combined with the vanadium leached from the uranium leach residues.

Cyclic Leaching - Ion Exchange Test

A cyclic leaching test was conducted through ten cycles. Three kilograms of ore were
leached, filtered and displacement washed; the solution passed through IRA-400 anion
exchange resin (1" dia. x 18" deep) with a retention time of ten minutes; the barren
effluent was used to pulp ore for the following cycle prior to acid addition for leach-
ing. The purpose of the test was to determine acid consumption and to what strength
vanadium could be allowed to build up without interfering. Table XIII on page 24 gives
the more important data concerning this test. Recoveries of ninety per cent or over
were obtained when the pH was one or below. In Cycle No. 6 the pH was held at 1.5 and
the recovery fell to 47.8 per cent. This cycle has not been included in computing the
overall averages. The overall recovery of vanadium was about 10 per cent and the leach
solution had built up to about 5 grams V$_2$O$_5$ per liter after 10 cycles. None of the
vanadium was adsorbed by the resin and there was no evidence that the increased vana-
dium concentration had any harmful effect. Filtration rates increased as the test pro-
gressed so there is no indication of a cyclic buildup of any impurity injurious to fil-
tration. Chloride content of the solutions had reached 9 grams Cl per liter at the
end of the tenth cycle. In actual operation this would be taken care of by a solution
bleed. This bleed would be in the form of uranium barren effluent from which the vana-
dium would be precipitated before discard. One test was made on this solution to
precipitate the vanadium. The solution was heated to 80°C, oxidized with sodium
chlorate and the acidity adjusted to pH 1.4 with NaOH. Eighty-four per cent of the
vanadium precipitated in a product assaying 76 per cent V$_2$O$_5$.

Table XIII

Data for Cyclic H$_2$SO$_4$ Leaching and Ion Exchange Test

(3 Kg. 17-comp. Ore each Cycle)

Cycle	H$_2$SO$_4$ - Lb per ton ore	per Lb U$_3$O$_8$ Extracted	Leaching Time Hours	pH	Wash ml H$_2$O	Relative Filtration Rate	% U$_3$O$_8$** Ore	% U$_3$O$_8$** Residue	U$_3$O$_8$ % Distribution Residue	Samples	Ppt	Ppt. Assay % U$_3$O$_8$
1	217	48	4	0.8	500	100***	0.251	0.026	10.5	3.2	86.3	92.0
2	207	41	4	0.8	1000	158	0.263	0.012	4.6	2.6	92.8	90.5
3	204	49	4	0.9	1000	145	0.221	0.011	5.0	5.3	89.7	90.5
4	204	42	4	0.9	1000	242	0.248	0.008	3.2	3.1	93.7	91.6
5	197	41	4	1.1	1000	180	0.252	0.013	4.8	4.2	91.0	87.6
6	173	66	4	1.5	1000	178	0.276	0.140	52.2	2.1	45.7	90.0
7	197	45	4	1.0	1000	185	0.240	0.022	9.3	4.2	86.5	91.6
8	197	46	4	1.0	1200	255	0.227	0.011	4.9	4.7	90.4	85.5
9	210	46	2	0.8	1100	280	0.251	0.024	9.7	4.3	86.0	91.3
10	197	42	2	0.9	1100	272	0.258	0.023	9.0	4.6	86.4	93.1
Avg.*	203	44	-	0.9	989	202	0.251	0.017	6.8	4.0	89.2	90.4

*   Cycle No. 6 not included

**   Calculated

*** Based on dry weight per unit area per unit time using first cycle as 100 - see section on filtration

Sulfuric acid consumption was about the same as in batch tests. However, there are other advantages to recycling. These are the elimination of possible uranium loss in the barren effluent, a more concentrated vanadium solution to precipitate and a saving in water.

Vanadium Leaching

Both 17-7 ore and uranium leach residue were given an oxidizing roast for 3 hours in an assay furnace muffle at 600°C and 800°C followed by a sulfuric acid leach (180 lbs H_2SO_4/ton of feed). Results of these tests appear in Table XIV below.

Table XIV

Roasting Tests

| Material | Roasting Temp. °C | Leach Liq. pH | % Extraction | |
			U_3O_8	V_2O_5
Ore	600	0.5	87.8	27.1
Ore	800	0.4	78.1	30.9
Residue	600	0.4	--	32.0
Residue	800	0.4	--	22.4

Extractions of uranium and vanadium were not particularly good although vanadium extraction improved. Uranium extraction was lowered by the high temperature roast. Another series of tests using reducing roasts (followed by H_2SO_4 leaching) were made at temperatures of 100°C, 400°C, 500°C, 600°C and 700°C in an atmosphere of illuminating gas. Another test involved a roast at 650°C in an atmosphere of SO_2. The results of these tests were poor.

A salt roast of the acid leach residue at 850°C, with 10 per cent by weight of NaCl being used, for 1 1/2 hours and followed by a water leach resulted in 52 per cent vanadium extraction. Further leaching, with 80 lbs. H_2SO_4 per ton of ore extracted 11 per cent more of the vanadium making the total extraction 63 per cent. The final residue was 0.29% V_2O_5.

A number of tests were made in which both ore and residue were mixed with varying quantities of concentrated H_2SO_4 and baked at 110°C for 18 to 65 hours, before leaching with water for 4 hours at 70°- 80°C and at 50 per cent solids. The results of these tests are tabulated in Table XV on page 26. The residue used was from a uranium leach in which about 95 per cent of the uranium and 10 per cent of the vanadium had been extracted by H_2SO_4 leaching.

Further work included baking at temperatures of 150°C, 200°C, and 300°C. The method consisted of mixing concentrated sulfuric acid (400 lbs./ton) with filtered uranium leach residue, and then placing the mixture in a furnace and baking at the indicated temperature for one-quarter to two-hour periods. After baking, sufficient water was added to form a 50% solids pulp, which was agitated for four hours at 70°-80°C. temperature. In one test, CaF_2 was added to the residue-acid mixture prior to baking. Results of these tests are shown in Table XVI on page 26. In the test marked "Blank", the uranium residue was agitated with 400 pounds of H_2SO_4 per ton at 50% solids, 70°-80°C., for four hours.

Table XV

Acid Baking Tests

Feed	H2SO4 Lbs./ton	Bake °C	Bake Hours	Leach Hours	Leach pH	Residue % V2O5	% Extraction U3O8	% Extraction V2O5
Res.	72	110	24	2	1.9	0.86	--	10.5
Res.	144	110	24	2	1.3	0.59	--	33.4
Res.	288	110	24	2	1.1	0.24	--	70.7
Res.	288	25	96	2	1.0	0.70	--	37.7
Res.	400	110	18	4	0.8	0.17	--	82.1
Ore	400	110	18	4	1.5	0.25	96.2	69.5
Ore	600	110	65	4	1.1	0.18	94.3	82.4

Note: Average V_2O_5 content of residues 0.9 per cent; ore 1.0 per cent.

Table XVI

The Effect of Time and Temperature on Vanadium Extraction

Baking Time, Hrs	°C	Final Residue %U3O8	Final Residue %V2O5	pH[1]	% Extraction[3] U3O8	% Extraction[3] V2O5
1	150	.0040	0.31	0.7	73.2	66.4
2	150	.0042	0.36	0.7	72.1	60.3
1	200	.0045	0.24	0.8	60.3	78.1
1[2]	200	.0052	0.28	0.8	68.3	72.9
2	200	.0053	0.44	0.8	69.0	64.7
1/4	300	.0063	0.30	0.9	58.6	71.2
1/2	300	.0068	0.37	1.1	60.3	67.7
Blank		.0011	0.48	0.7	91.9	55.3

1/ pH of leach liquor
2/ 100 pounds CaF_2 added per ton of feed.
3/ Extraction from residue

Note: Test feed .014% U_3O_8, 0.99% V_2O_5 average.

When these results are compared with those in Table XV, baking at 110°C for 18 hrs. produced the best extraction with respect to vanadium (82.1% vanadium extraction, residue assay 0.17% vanadium). The test using CaF_2 was made to determine whether HF, produced by the interaction of CaF_2 and H_2SO_4, would aid in opening up the vanadium bearing mineral. Judging from the results obtained, the addition of CaF_2 was not helpful. The data above also indicate that at any baking temperature, increased time lowers vanadium extraction.

The results show that by the use of sufficient H_2SO_4 (300 to 400 pounds per ton), 70 to 80% of the vanadium in the uranium leach residue can be extracted using the sulfation technique, plus a low temperature bake followed by water leaching. These results were better than those obtained by the normal practice of salt roasting at 800°C followed by a water leach, where vanadium extraction was 52%. This work was done on a composite of the 17 - 1-6 ore samples.

One sample of low lime (Wild Steer) ore assaying 0.79% CaO and containing about 0.37% U_3O_8 and 3.4% V_2O_5 was tested to determine how such an ore would respond to an acid-baking treatment. Sulfuric acid and ore were mixed so that the entire amount of ore was wet. For the lower amounts of acid it was necessary to use dilute rather than concentrated H_2SO_4 to accomplish this. Following this the mixture was baked overnight at 90° to 100°C. The water leach was carried on at 70°C and 50% solids for four hours. The results are quite similar to those obtained with the Lukachukai ores if allowance is made for acid required to neutralize the lime. Table XVII which follows summarizes the results of the tests.

Table XVII

Acid-Bake Leaching Tests of Wild Steer Ore

Test No.	1	2	3	4	5	6
Lb H_2SO_4/T Ore	100	200	300	400	500	600
Temp bake, °C	90	90	90	90	90	90
Time, hrs	18	18	18	18	18	18
Water Leach Temp, °C	70	70	70	70	70	70
Water Leach Time, hrs	4	4	4	4	4	4
Water Leach pH	3.2	1.6	1.2	0.8	0.6	0.5
% Extraction U_3O_8	89.5	98.8	97.4	97.7	97.6	97.6
% Extraction V_2O_5	59.0	67.3	81.6	86.7	94.0	95.3
Calc Head Assay U_3O_8	0.48	0.41	0.38	0.39	0.45	0.37
V_2O_5	2.9	3.4	3.0	3.1	3.3	3.4

For comparison one test was run in which the ore was leached at pH 1 for four hours at room temperature and 50 per cent solids. This required 42 pounds of H_2SO_4 per ton of ore and resulted in a 94.5% uranium extraction and a 44.1% vanadium extraction.

Vanadium Precipitation

To determine the feasibility of precipitating vanadium "red cake" from acid leach liquors a test was run on uranium barren solution assaying 3.35 grams V_2O_5 per liter. One hundred and fifty ml of this solution was agitated at 80°C for 3 hours with one-half gram of $NaClO_3$. The pH was adjusted to pH 1.2 - 1.4 with NaOH. Eighty-five per cent of the vanadium precipitated in a product assaying 76.3% V_2O_5. This assay was on a sample oven dried at 110°C.

Leach liquors from treating the uranium leach residues were also precipitated. The solution was prepared by H_2SO_4 leaching ore, removing uranium by anion exchange, extracting additional vanadium by baking the residue with concentrated H_2SO_4 followed by leaching with the uranium barren solution for additional vanadium extraction. Part of this leach liquor was bled for vanadium precipitation and the remainder returned to start a new cycle (see section on Flowsheets Tested). Precipitation results for eight cycles of this procedure are shown in Table XVIII on the following page.

Table XVIII

Vanadium Precipitation From Cyclic Test of Flowsheet No. 2

	Grams V_2O_5 Per Liter		Per Cent[1]	Ppt. Assay[2]
Cycle	Feed Soln	Spent Soln	Precipitation	% V_2O_5
1	0.8	-	-	-
2	4.7	3.8	19	84.5
3	5.8	0.8	86	71.2
4	8.8	0.9	90	80.1
5	9.9	0.9	91	79.2
6	9.8	3.4	65	83.7
7	14.4	2.6	82	76.5
8	12.3	2.8	77	77.4

[1] Calculated from solution assays only
[2] Oven dried at 110°C

Filtration and Washing of Residues

A. Relative Filtration Rates

The leach residues from all of the cyclic tests were checked for filterability.
This was done by forming a filter cake for three minutes on a standard 0.1 square
foot filter test leaf and washing thoroughly. The dry weight of the cake from the
first test run was assigned a value of 100. The weight of this cake was 59.5 grams,
so multiplying the weight of all subsequent test cakes by $\frac{100}{59.5}$ or 1.681 gave a factor
for comparing the relative filterability of the various pulps. For example a rela-
tive filtration rate of 200 would mean that for the conditions of the test the fil-
tration rate (units of weight per unit of area per unit of time) was twice that of
the original standard. In this way, the effects on filterability of the various
methods of treatment were compared.

B. Determination of Filter Requirements

Almost all of the test work was done on a composite of the first shipment of ore
(17-1-6). The second shipment (sample 17-7) was more filterable by a factor of over
five. Some work was done with this material to determine filter requirements for
leaching and washing two hundred tons of ore per day. A quantity of pulp was made
by leaching 4 kg. of ore at 50% solids with 260 pounds of H_2SO_4 per ton of ore for
four hours at room temperature. This pulp was used to conduct the various tests to
be discussed. First, test cakes were formed for various times to determine the weight
of solids and cake thickness obtained . The results are shown in Table XIX on the
following page.

Table XIX

Solids Filtered From Leach Pulps Using Various Times

Form Time Secs	Grams Cake Dry	Thickness Inches	Lbs/sq. ft. of Filter Area
15	101.7	1/2	2.24
30	133.5	3/8	2.94
45	163.0	5/8	3.59
60	204.5	3/4	4.50
120	297.7	1 1/16	6.56

For the purpose of estimating rotary filter requirements the filter cycle shown in Figure 9 was assumed.
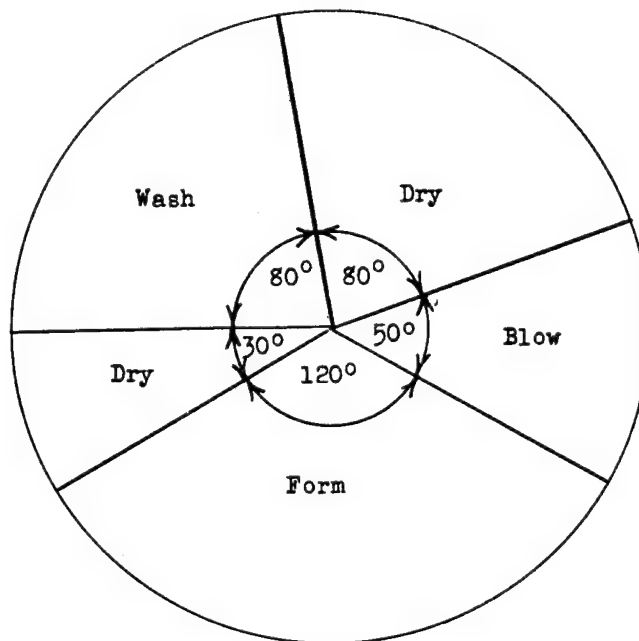
Figure 9.   Assumed Filter Cycle

Leach pulp was filtered using this cycle for the various times indicated as being reasonable by the data in Table XIX. Forming times greater than 60 seconds were not tested as the cake was so thick that it would probably fall off the filter prematurely and certainly would not wash. Table XX shows the results of the tests using a 0.1 square foot test leaf at 27 inches Hg pressure. Table XX appears on the following page.

Table XX

Results of Testing Various Filter Cycle Times

	Degrees	Per Cent of Total	60 Sec. Cycle	120 Sec. Cycle	180 Sec. Cycle
Form	120	33.3	20	40	60
Dry	30	8.3	5	10	15
Wash	80	22.2	13.5	27	40
Dry	80	22.2	13.5	27	40
Blow	50	14.0	8	16	25
Total	360	100.0	60	120	180
Cake Thickness-inches			1/4	1/2	9/16
Grams Wet Cake			134	175	219
Grams Dry Cake			100	130	162
% Moisture			25.4	25.7	26.0
Ml Filtrate & Wash			120	175	220
Cake Assay					
% Total U_3O_8			0.06	0.06	0.07
% H$_2$O Sol. U_3O_8			0.04	0.04	0.05
Indicated Number of Pounds of Residue Per Square Foot of Filter Area Per Day.			3170	2061	1713

C. Washing Residues

The results in Table XX show that proper washing would not be possible if attempted on a drum filter in a single step filtration. Water soluble uranium in the final residue was 0.04 - 0.05% which, in the case of this ore, represents almost 15 per cent of the total.

Further tests were conducted in which cakes formed for various times were displacement washed for times longer than those allowed by the cycle in Table XX. Figure 10 shown on the following page gives the results of these washing tests. These indicate that the water necessary is equal to 75 - 100% of the dry weight of residue washed. The data also show that the thicker cakes wash somewhat more efficiently.

D. Filtration of Sodium Carbonate Leach Pulps

The pulps from acid leaches were homogeneous. While there were rather wide variations in filterability indicated all of the samples tested lent themselves within reason to filtration and displacement washing. Sodium carbonate pulps, however, classified into a sand and slime fraction. While the sand portion was easily filtered, the slime was not. This condition would make filter operation difficult.

Filter leaf tests showed acid leach pulps to be about five times as filterable as carbonate pulps. The data appears in Table XXI on the following page.

Figure 10. Washing Characteristics of Acid Leach Pulps
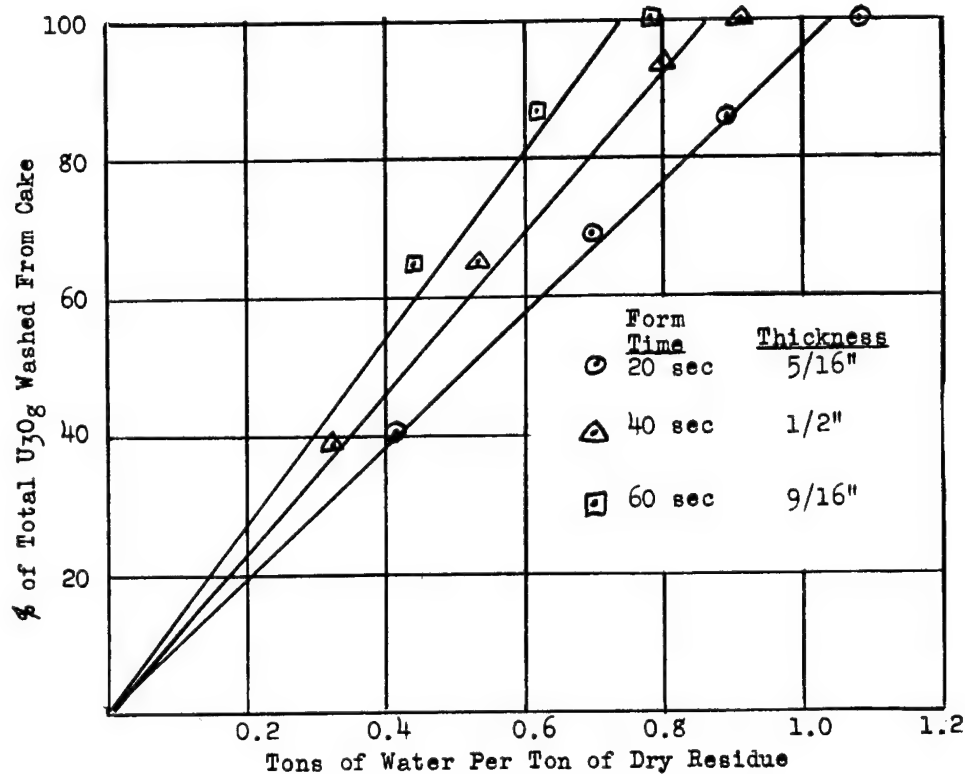
Table XXI

Comparison of Acid and Carbonate Filtration

(3 min. form time - 0.1 sq. ft.)

Kind of Pulp	H_2SO_4	Na_2CO_3	Na_2CO_3
Temp ^oC	25	25	80
grams cake wet	222	35.5	43.0
grams cake dry	165	26.5	33.5

Note: Carbonate leach 150 lbs. Na_2CO_3 per ton of ore -
Acid leach 250 lbs. H_2SO_4 per ton of ore.

Flowsheets Tested

Four flowsheets were tested to determine their usefullness as a method for recovering uranium and vanadium from the ore. Each was run for the number of cycles indicated and will be discussed separately.

Conditions for ion exchange were the same in all cases. That is a one inch diameter column containing 200 ml of wet settled resin was used. Retention time of solutions in the resin bed (assuming 40% voids) was ten minutes. Elution of the uranium from the column was done with 0.1N HCl - 0.9N NH$_4$Cl. Following elution the solution was neutralized to pH 7 with NH$_3$. After removal of the uranium precipitate by filtration the filtrate was acidified with HCl to pH 1.0 and was ready for reuse for elution.

HCl used, amounted to 3.4 pounds per pound of U$_3$O$_8$ and NH$_3$ to 1.6 pounds per pound of U$_3$O$_8$. These figures are conservative as the column was somewhat oversize and consequently not loaded to its full capacity. The preliminary ion exchange data indicated that resin loadings about twice that obtained would be possible. If this were the case the HCl and NH$_3$ used would be one-half the above figures.

A. Flowsheet No. 1

Here ore was leached and filtered; the solution ion exchanged to remove uranium and recirculated to leach another batch of ore. A portion of the barren solution was sent to a second leaching section where the residue of the first leach was leached with strong hot H$_2$SO$_4$ to extract vanadium.

The expected advantage of the two step leaching procedure over a single hot leach was to have a dilute acid uranium bearing solution to ion exchange and a second liquor of high acid strength to give maximum vanadium extraction. All of the makeup acid went to this vanadium leach and was to be regulated to give sufficient residual acid strength in the filtrate to leach the uranium in the ore. This flowsheet is shown in Figure 11 appearing on page 34 and the operating data in Table XXII on page 35.

Two thousand grams of ore were leached each cycle. Leaches were mechanically agitated 2 hours at room temperature for the uranium extraction step, and 4 hours at 90°C for the vanadium leach. Except for the starting cycle, the H$_2$SO$_4$ was added to the vanadium leach, and the amount was sufficient so that the residual free acid was adequate to leach the uranium from the incoming ore. The amount of acid used was limited by the pH of the uranium leach. When this was too low (pH less than 1), the column effluents were high in uranium because of lowered uranium capacity of the resin. On the other hand, if the acid was reduced to a point where the uranium leach was at pH 1, the amount of acid was below the amount necessary to leach a reasonable amount of vanadium.

B. Flowsheet No. 2

After seven cycles, a change was made to Flowsheet No. 2. Here it was possible to use more acid and still maintain about a pH 1.0 feed to the ion exchange column. This was attributed to the more complete reaction of acid and residue as a result of the baking procedure. With the increase in acid consumption, came better extraction of vanadium. This flowsheet was operated for eight cycles. Recoveries of uranium were about the same for both procedures.

This flowsheet is shown in Figure 12 on the following page and the operating data given in Table XXII. In both Flowsheet 1 and 2 there was a buildup of salts. These were principally $K Al(SO_4)_2 \cdot 12 H_2O$ which precipitated from solutions on cooling following the hot acid leach for vanadium recovery. This condition would undoubtedly cause trouble in filtration of either or both of the uranium and vanadium leach residues as well as interfere with the passage of solutions through the ion exchange columns which in effect are filter beds.

C. Flowsheet No. 3

This is shown in Figure 13 on page 36.. Here the uranium and vanadium circuits were kept isolated with the result that salt buildup was lower in the uranium circuit (3.6° Bé max.), uranium precipitate grade was better and the uranium leach filtration satisfactory. Data for this test are presented in Table XXXIII on page 38.

This flowsheet is the procedure recommended for uranium recovery and the principal shown of keeping the uranium and vanadium circuits isolated should be followed if a vanadium recovery step is added, whether it be by acid baking or salt roasting.

D. Flowsheet No. 4

This flowsheet shown in Figure 14 on page 37 with operating data in Table XXIII was tested to determine if both uranium and vanadium could be extracted from the ore simultaneously using an acid bake. Extractions of both metals were excellent, but even though the operation was not cyclic it was plagued with crystallization of salts which made filtration of leach residues unusually erratic and column operation difficult.
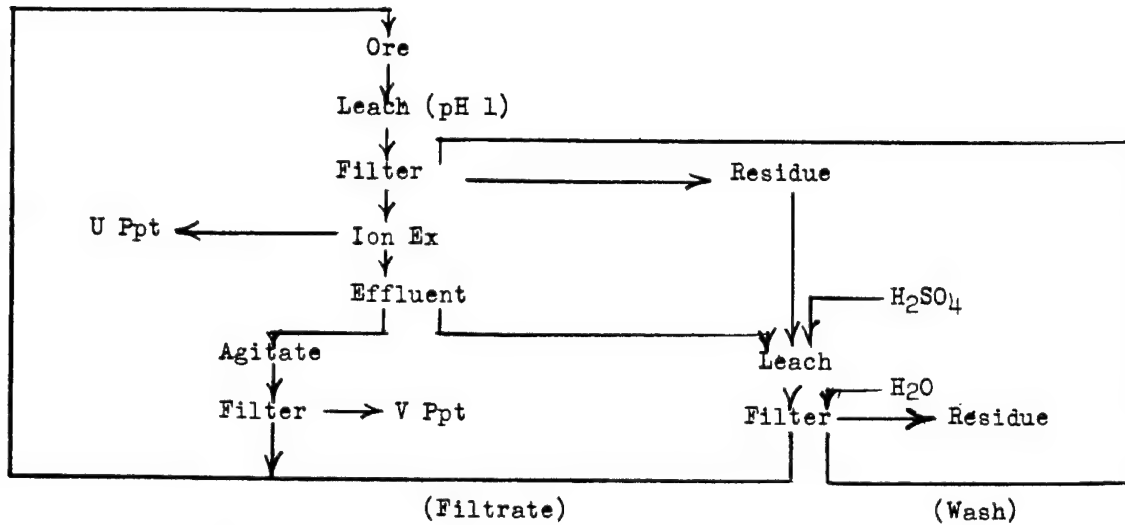
Using Strong Acid Leach For Vanadium Extraction

Figure 11.   Lukachukai Ore Flowsheet No. 1 (Simplified)
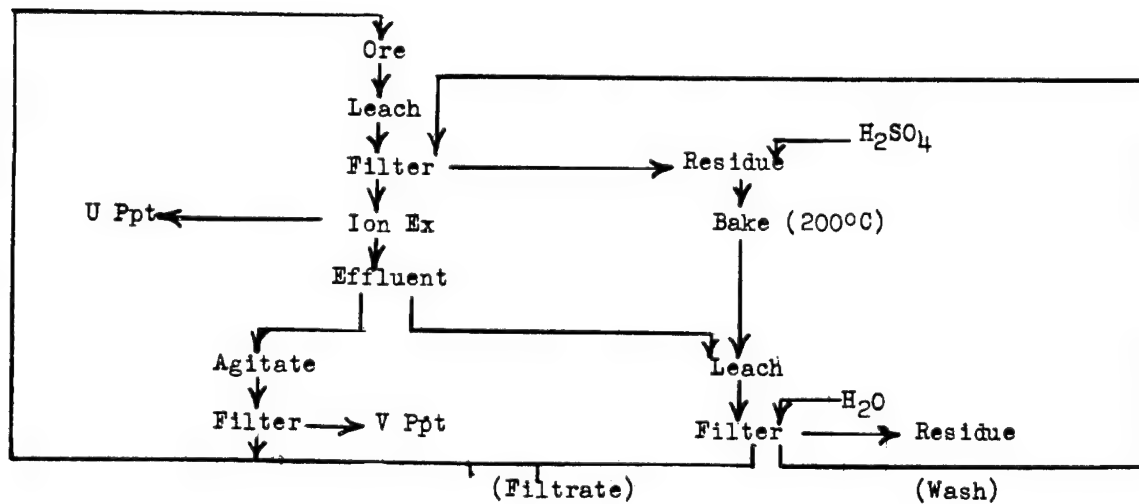
Using Strong Acid Bake for Vanadium Extraction

Figure 12.   Lukachukai Ore Flowsheet No. 2 (Simplified)

Table XXII

Data For Leaching and Ion Exchange Tests 17 – 1-6 Ore

Cycle	H₂SO₄ Lb/T Ore	U Leach Soln pH	U Leach Soln °Be	g U₃O₈/L Leach Soln	g U₃O₈/L Col. Eff.	g U₃O₈ Adb.	Relative[1] Filtration Rate U Leach	Relative[1] Filtration Rate V Leach	Calc %U₃O₈ Ore	Residue Assay %U₃O₈	Residue Assay %V₂O₅	Ppt Assay %U₃O₈
Flowsheet No. 1												
1	634	-	5.1	1.90	0.05	4.63	200	-	-	0.001	0.18	81.3
2	520	-	11.7	1.40	0.22	3.19	-	-	-	0.004	0.18	79.4
3	510	-	18.3	1.80	1.20	1.57	-	-	-	0.007	0.12	68.0
4	381	0.5	23.1	2.30	1.30	2.05	49	255	-	0.026	0.22	63.1
5	300	0.9	24.4	4.18	1.07	5.04	101	173	-	0.024	0.31	69.5
6	407	0.6	22.6	4.00	1.40	4.34	80	155	-	0.020	0.31	73.1
7	340	0.7	22.8	3.60	-	-	78	152	-	0.011	0.31	-
Avg.	442	-	-	-	-	3.47	-	-	0.224	0.014	0.24	72.8
Flowsheet No. 2												
1	600	1.4	2.1	2.40	0.00	4.44	237	313	-	0.004	0.10	74.7
2	476	0.8	13.6	2.70	0.21	4.18	85	232	-	0.028	0.10	77.8
3	579	0.8	15.7	3.25	0.05	5.63	84	223	-	0.044	0.09	72.2
4	450	1.2	21.5	3.00	0.04	5.30	82	345	-	0.016	0.09	82.0
5	519	1.1	18.8	2.81	0.03	4.25	105	133	-	0.017	0.09	69.0
6	539	1.1	20.8	2.12	0.06	3.93	94	200	-	0.008	0.08[2]	72.3
7	427	1.0	27.1	3.21	0.24	5.64	24	119	-	0.010	0.08	66.4
8	503	1.2	22.6	2.70	0.06	4.33	66	168	-	0.003	0.08	86.5
Avg.	512	-	-	-	-	4.71	-	-	0.246	0.016	0.09	74.0

Note: 2000 grams ore leached each cycle. Ion exchange conditions were as follows:
200 ml wet settled IRA-400 resin
Retention time during exhaustion --10 minutes
Retention time during elution --10 minutes
Eluting solution was 0.9M NH_4Cl – 0.1M HCl – The eluate after precipitation
with ammonia was made up to acid and salt strength and recycled.

1/ See section on Filtration
2/ Value estimated – Analyses not available
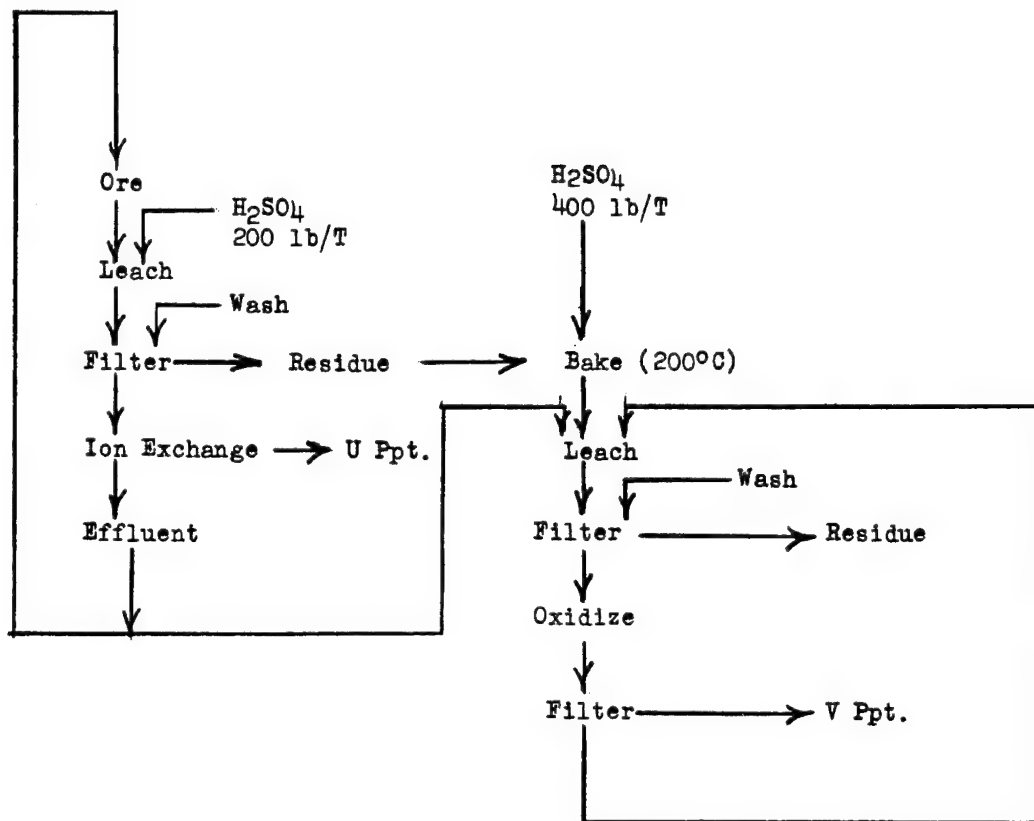
Figure 13. Condensed Flowsheet No. 3 (Simplified)
Using Acid-Bake of Residue Without Recirculation of V Leach Solution
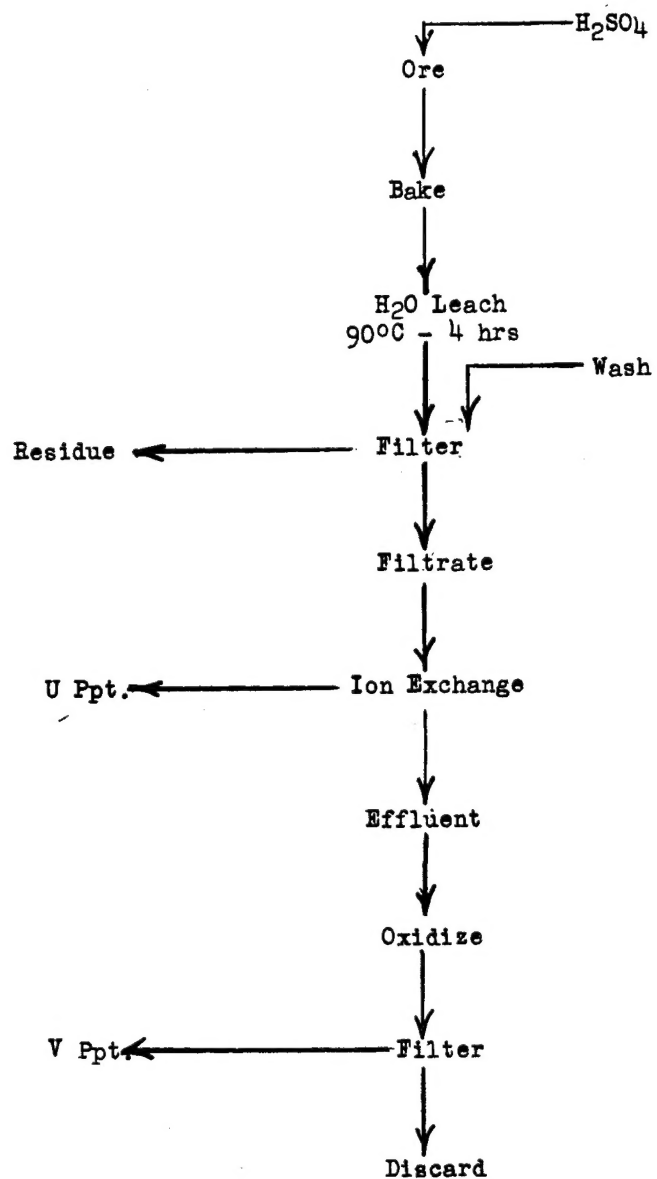
H$_2$SO$_4$

Ore

Bake

H$_2$O Leach
90°C - 4 hrs

Wash

Residue ◄——— Filter

Filtrate

U Ppt. ◄——— Ion Exchange

Effluent

Oxidize

V Ppt ◄——— Filter

Discard

Figure 14.   Condensed Flowsheet No. 4 (Simplified)
Using Acid-Bake of Whole Ore

Table XXIII

Data For Leaching and Ion Exchange Tests 17 - 1-6 Ore

	Cycle	H$_2$SO$_4$ Lb/T Ore	U Leach Soln pH	U Leach Soln °Be	g U$_3$O$_8$/L Leach Soln	g U$_3$O$_8$/L Col. Eff.	g U$_3$O$_8$ Adb.	Relative[3] Filtration Rate U Leach	Relative[3] Filtration Rate V Leach	Calc %U$_3$O$_8$ Ore	Residue Assay %U$_3$O$_8$	Residue Assay %V$_2$O$_5$	Ppt Assay %U$_3$O$_8$
Flowsheet No. 3	1	210	1.1	2.4	1.59	0.01	3.87	176	49	--	0.04	0.13	87.2
	2	210	1.0	2.3	1.71	--	4.20	138	36	--	0.01	0.09	86.0
	3	198	1.3	2.1	1.79	0.01	4.52	138	-	--	0.06		84.1
	4	208	1.2	2.5	1.93	0.05	5.13	71	66	--	0.02	0.07	86.6
	5	210	1.1	2.6	1.71	0.04	4.86	90		--	0.04		90.8
	6	212	1.1	2.9	1.59	0.03	3.48	72		--	0.02		83.4
	7	214	1.2	3.0	1.82	0.16	4.90	102	45	--	0.03	0.03	80.9
	8	206	1.4	2.9	1.76	0.06	3.60	117		--	0.06		79.2
	9	225	1.2	3.6	2.55	0.03	6.12	113	32	--	0.03	0.07	80.9
	Avg.	210	-	-		0.05	4.53	113	45	0.31	0.03	0.07	84.3
Flowsheet No. 4	1	600	1.1[2]	11.5	1.59	0.01	5.10	98	-	--	0.01	0.08	86.7
	2	600	1.1	11.8	1.72	0.01	5.20	162	-	--	0.02	0.07	80.3
	3	600	1.1	13.7	1.60	0.01	5.02	33	-	--	0.02	0.06	81.3
	4	600	1.1	16.8	2.38	0.01	5.74	36	-	--	0.02	0.07	82.9
	5	600	1.1	18.1	2.30	0.01	5.20	55	-	--	0.02	0.14	79.2
	6[1]	600	1.1	17.3	2.61	0.01	6.11	135	-	--	0.02	0.09	77.3
	7[1]	600	1.1	17.3	2.07	0.01	4.66	223	-	--	0.02	0.09	82.0
	8[1]	600	1.1	17.9	2.43	0.01	5.77	184	-	--	0.02	0.10	80.1
	Avg.	600	-	-		0.01	5.35	116	-	0.26	0.02	0.09	81.2

Note: 2000 grams ore leached each cycle. Ion excahnge conditions were as follows:
200 ml wet settled IRA-400 resin
Retention time during exhaustion --10 minutes
Retention time during elution --10 minutes
Eluting solution was 0.9M NH$_4$Cl - 0.1M HCl - The eluate after precipitation
with ammonia was made up to acid and salt strength and recycled.

1/ 17-7 ore used. All other cycles 17-1-6 comp. used
2/ About 50 grams free H$_2$SO$_4$ per liter at end of leach neutralized to pH shown
3/ See section on filtration

Precipitate Grade

Two grades of precipitates were produced. In the case of Flowsheet No. 3 where uranium recovery was isolated from any attempt to recover vanadium other than the 10 - 20 per cent soluble under the uranium leach conditions the grade was higher.

Table XXIV shows assay of precipitates.

Table XXIV

Assay of U Precipitates

	U_3O_8	P_2O_5	V_2O_5	Fe
U Circuit Isolated	92.0	2.40	0.85	0.93
With recycled V solutions	81.0	1.36	3.5	3.17

Table XXV on the following page gives results of a spectrographic analysis of the two products. The lower grade is due to additional impurities taken into solution by the more rigorous leaching conditions necessary to solubilize the vanadium in sulfuric acid.

Table XXV

Spectrographic Analysis of Precipitates

Element	90% U_3O_8 Ppt	70% U_3O_8 Ppt
Al	0.1 - 0.001	0.01 - 1.0
Sb	nil	nil
As	<0.01	<0.01
Be	nil	nil
Bi	nil	nil
B	<0.01	0.1 - 10
Cd	<0.01	<0.01
Ca	<0.01	<0.01
Cr	<0.01	<0.01
Co	nil	nil
Cu	<0.01	<0.01
Ga	nil	nil
Ge	nil	nil
Au	<0.01	<0.01
Fe	0.01 - 1.0	1.0 - 100
La	nil	nil
Pb	<0.01	0.001 - 0.1
Mg	<0.001 - 0.1	0.01 - 1.0
Mn	<0.01	<0.01
Hg	<0.01	<0.01
Mo	nil	nil
Ni	<0.01	nil
Nb	nil	nil
Pd	nil	nil
P	0.1 - 10	<0.01 - 1.0
Pt	nil	nil
Si	0.1 - 10	0.1 - 10
Ag	<0.01	<0.01
Na	<0.01	0.001 - 0.1
Ta	nil	nil
Sn	nil	nil
Ti	<0.01	0.01 - 1.0
W	<0.01	<0.01
U	1.0 - 100	1.0 - 100
V	0.001 - 0.1	0.01 - 1.0
Zn	<0.01	0.001 - 0.1
Zr	nil	nil

Note: Figures given are in per cent.